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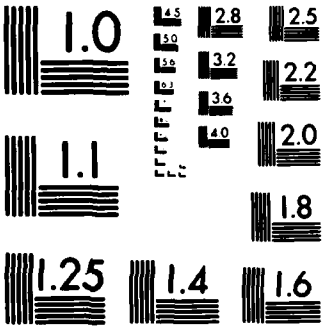

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AFWAL-TR-84-4160

COMPOSITIONAL AND STRUCTURAL ANALYAIS OF AIR FORCE MATERIALS

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11 January 1985

FINAL REPORT FOR PERIOD 15 May 1982 - 15 September 1984

Approved for public release: distribution unlimited.

MATERIALS LABORATORY
AIR FORCE WRIGHT AERONAUTICAL LABORATORIES
AIR FORCE SYSTEMS COMMAND
WRIGHT-PATTERSON AIR FORCE BASE, OHIO 45433

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This technical report has been reviewed and is approved for publication.

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Work performed during the period 15 May 1982 - 15 September 1984 in support of various Air Force programs is summarized. The work covers routine analyses, methods development, support programs, service functions, materials characterization and the application of various analytical chemical techniques. Analytical techniques included:

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FOREWORD

This report was prepared by Technical Services Group, Fairborn, Ohio and MONSANTO, Dayton Laboratory, Dayton, Ohio. The work described in this report was administered under the direction of the Materials Laboratory, Air Force Systems Command, Wright-Patterson Air Force Base, Ohio, as Contract No. F33615-82-C-5028. Mr. James H. Muntz served as Project Engineer.

This work was conducted at the Materials Laboratory, Wright-Patterson Air Force Base, Ohio and at the Dayton Laboratory of MONSANTO, Dayton, Ohio, during the period 15 May 1982 - 15 September 1984. This report was submitted in draft form in October 1984.

and High Pressure Liquid Chromatography (HPLC);

TABLE OF CONTENTS

Originator Supplied Keywords include:

SECTION I

INTRODUCTION

This report was prepared by Technical Services Group, Fairborn, Ohio and MONSANTO, Dayton Laboratory, Dayton, Ohio and is the final report of USAF Contract F33615-82-C-5028. This report covers work performed on the above contract from 15 May 1982 through 15 September 1984 at the Materials Laboratory, Wright-Patterson AFB, Ohio and at MONSANTO's Dayton Laboratory, Dayton, Ohio.

MONSANTO was the prime contractor for this program. Technical Services Group was a subcontractor to MONSANTO from 16 July 1983 through 15 September 1984.

Dr. A. D. Snyder, Dr. W. H. Hedley, and Mr. F. N. Hodgson were involved in the management of this contract for MONSANTO. Mr. J. Workman was the Principal Investigator.

The individuals responsible for the bulk of the analyses conducted on this contract were Mr. Donald Douglas, Mr. Michael Jubara, Mr. Thomas Kerschner, Mr. Thomas Stout, and Dr. Chi Yu. A portion of the work performed on this program required unique analytical capabilities and/or facilities and was conducted by commercial and university laboratories.

Sections II through X discuss the work performed at Wright-Patterson. Section XI discusses work that was performed by other laboratories.

SECTION II

ANALYTICAL SUPPORT

Table 1 shows the number of samples received each month for analysis and illustrates the support provided to the various branches of the Materials Laboratory over the course of this program. Samples received from Air Force organizations outside of the Materials Laboratory are shown as "OTHER" samples.

TABLE 1

USAF Contract F33615-82-C-5028

Samples Received for Analysis
(by organization)

	MLL	MLP	MLB	MLS	OTHER	TOTAL
15-31/5/82	55	0	37	8	13	113
Jun	40	2	75	27	10	154
Jul	33	2	134	60	23	252
Aug	39	0	178	53	21	291
Sep	70	11	92	21	10	204
Oct	50	0	83	36	20	189
Nov	39	10	67	44	11	171
Dec	16	2	131	21	10	180
Jan 83	46	1	119	16	69	251
Feb	52	0	76	18	4	150
Mar	31	1	124	95	41	292
Apr	21	0	78	57	26	182
May	18	9	107	50	26	210
Jun	68	3	95	21	44	231
Jul	106	3	116	34	0	259
Aug	78	0	119	54	13	264
Sep	88	9	96	49	45	287
Oct	80	11	134	39	11	275
Nov	45	25	87	43	14	214
Dec	38	11	83	27	18	177
Jan 84	19	6	110	21	12	168
Feb	26	0	108	110	19	263
Mar	25	6	182	112	4	329
Apr	32	2	101	185	9	329
May	9	16	86	113	24	248
Jun	8	0	157	59	22	246
Jul	42	5	60	120	11	238
Aug	86	11	111	54	8	270
1-15 Sep	33	2	42	46	12	135
Totals	1293	148	2988	1593	550	6572

SECTION III

MICROCHEMICAL ANALYSES

Microchemical analyses required a substantial and continuing effort during the course of this program.

The determination of carbon, hydrogen, and nitrogen in organic materials represents the most frequently requested technique in the area of microchemical analyses. Early in the program, the Air Force received a new Perkin-Elmer Model 240-C carbon, hydrogen and nitrogen analyzer with a dedicated data station.

The arrival of this instrument permitted the conversion of the existing Perkin-Elmer Model 240-A instrument to use for the analysis of oxygen in organic materials.

Determinations of a variety of elements in organic materials were conducted on a regular basis including analyses for halogens, sulfur, silicon, and phosphorus.

During the course of the program a variety of wet chemical analyses were conducted on a one-time or irregular basis including the determination of weight loss upon ignition, ash content, density, and extractable solids.

Methods were developed or adapted for use in the determination of sulfur in arsenic-germanium sulfide films.

SECTION IV

MASS SPECTROMETRY

The mass spectrometry effort during the course of this program was limited to the use of the Finnigan 4021 GC/MS/DS system. While the DuPont 21-490 system was available for use, it was not required during this program.

Several improvements in the Finnigan system were implemented during this program. An ion source upgrade kit was installed to minimize the time required in the periodic cleaning of the ion source and in the conversion from one ionization mode to another. This upgrade included a modification to the gas chromatograph to permit subambient temperature programming by liquid nitrogen cooling. A new revision of the system software was acquired and installed. A copy of the National Bureau of Standards library of mass spectral data for selected organic compounds was acquired on disk for use as a reference library on the new system.

Samples received for mass spectrometric analysis included starting materials, intermediates, and reaction products from the various branches involved in synthesis work, silicon based lubricating and hydraulic fluids, and a variety of environmental samples.

Substantial gas chromatography/mass spectrometry support was provided to MLBT in the characterization of the degradation products of candidate hydraulic fluids following thermal and pressure stressing. This support required the analysis of headspace and liquid samples using programmed temperature capillary column gas chromatography/mass spectrometry.

SECTION V

MOLECULAR SPECTROSCOPY

The molecular spectroscopy support provided over the period of this contract included infrared as well as UV-visible spectroscopy.

Infrared spectroscopy (IR) was used on a continuing basis as a primary and supporting analysis technique. The most frequent requests for IR support originated with the various organizations involved in synthesis work. IR was used in the identification of coatings, deposits, structural materials, and electrical insulators.

In the second year of the contract, substantial support was provided in the use of infrared spectroscopy in the pre- and post-test phases of the rain erosion testing of optical coatings. This work was in support of several studies conducted within the Materials Laboratory to investigate the changes in IR transmission characteristics of coated optical materials following exposure to a rainfield. Infrared data were obtained for approximately 100 coated optical plates before and after rain erosion testing.

A screening method for the determination of asbestos materials by infrared spectroscopy was adapted for use as a supporting technique. This method was found to be quick and to provide identification of asbestos type insulation with great reliability. While x-ray diffraction (XRD) analysis remains the definitive analysis method for asbestos, XRD work can be performed at a maximum rate of only two samples per day. The use of IR as a supporting technique in asbestos analyses permits a quick screening of samples to provide preliminary determination of asbestos presence and type and to aid in the prioritization of samples for subsequent analysis by XRD.

During the course of this program, the Air Force acquired a new Perkin-Elmer Lambda 3 UV-visible instrument. This instrument is configured for use with the Perkin-Elmer data station. At present the Lambda 3 instrument shares a data station with the Perkin-Elmer Model 683 Infrared instrument. Since the two instruments are infrequently in use at the same time, this arrangement has permitted the maximum utilization of the data station.

SECTION VI

ATOMIC SPECTROSCOPY

The requirements for support in the area of atomic spectroscopy included work in atomic absorption as well as in emission spectroscopy.

Atomic absorption analyses were provided on a continuing basis. This work included analyses to support alloy typing for various materials as well as confirmation of composition for novel materials being prepared with the Materials Laboratory.

Support in the area of emission spectroscopy was limited to the continued operation of the instrument with trained personnel during periods of leave or illness of the primary government analyst.

SECTION VII

SEPARATIONS

The requirement for analyses using gel permeation chromatography (GPC) and high pressure liquid chromatography (HPLC) was constant and high during the course of this program.

GPC analyses were conducted routinely for the determination of molecular weight distribution in polymers. This work was performed almost exclusively for MLBT in support of their work in the synthesis and characterization of FASIL materials.

Initially, these GPC analyses were conducted using a suite of polystyrene standards and yielded molecular weight data relative to polystyrene. To overcome the inherent limitations of this "polystyrene relative" data, a FASIL-specific GPC calibration technique was conceived and implemented. A limited number of FASIL samples were submitted for fractionation and fraction collection by GPC.

GPC analyses were also conducted on samples of epoxy oligomers.

HPLC analyses were conducted on a variety of samples during the course of this program. An HPLC method for the analysis of phenols in paint stripper was developed.

Separations were also conducted using classical liquid-liquid and Soxhlet techniques. These separations were performed to assess the percentage of extractable material in bulk samples and as sample preparation steps for other analyses.

SECTION VIII

POWDER X-RAY DIFFRACTION

The routine use of powder x-ray diffraction (XRD) was, for the most part, limited to the analysis of insulation materials for asbestos content.

Nonroutine XRD work included the analysis of a substantial number of samples associated with a study of turbine blade contamination, the characterization of several arsenic-germanium sulfide samples, and the analysis of small diameter alloy wires.

SECTION IX

GENERAL SUPPORT

General analytical work was conducted on a continuing basis over the course of this program. Within the area of general support, the determination of carbon in steel represents the most frequently requested analysis.

Other work conducted in the area of general support includes the following analyses: the determination of sulfur, silicon, and chlorine in metals, isocyanate, conductivity, and water hardness. A polarographic method for the determination of copper and palladium catalyst residues in polymer starting materials was investigated.

SECTION X

EMERGENCY SAMPLES

In many respects the timely and accurate analysis of emergency samples represents the most interesting and important work performed during the course of this program. Unfortunately, the nature of this work precludes its discussion within this report.

SECTION XI

SUBCONTRACTED ANALYSES

A number of analyses were conducted by outside laboratories during the course of this program. These analyses required specialized instrumentation or expertise not available within the Materials Laboratory.

Subcontracted analyses included carbon-13, proton, fluorine-19, and silicon-29 nuclear magnetic resonance studies, the determination of residual gases in semiconductors, spark source mass spectrometric assay of alloys, ESCA and Auger analyses of surfaces, identification of materials by optical microscopy, and the analysis of elastomeric surfaces using fourier transform infrared spectroscopic. Single crystal x-ray analyses were used to elucidate the structure of several new compounds. Several samples were analyzed using coupled size exclusion chromatography/low angle laser light scattering to determine weight average molecular weight distributions.

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